NIST's Advanced Technology Program

New Developments in Membrane Technology R&D

The 13th Annual Meeting of the North American Membrane Society

Long Beach, California, May 11-15, 2002

Robert A. Beyerlein and Gerald P. Ceasar
National Institute of Standards and Technology
100 Bureau Drive Stop 4730
Gaithersburg, MD 20899-4730
http://www.atp.nist.gov

ATP is part of NIST

NIST Mission:
Strengthen the U.S. economy and improve the quality of life by working with industry to develop and apply technology, measurements, and standards.

- **3,300 employees**
- **$800 million annual budget**
- **1,200 industrial partners**
- **2,000 field agents**
- **1,550 guest researchers**
- **$1.8 billion co-funding of industry R&D**
- **National measurement standards**

Helping America Measure Up

NIST Assets Include:

Advanced Technology Program

Partnership with private industry to accelerate the development of high-risk, enabling technologies with broad benefits for the entire economy and society.

Measurements and Standards Laboratories

Nation's ultimate reference point for measurements, standards, and technology research to support industry, science, health, safety, and the environment.

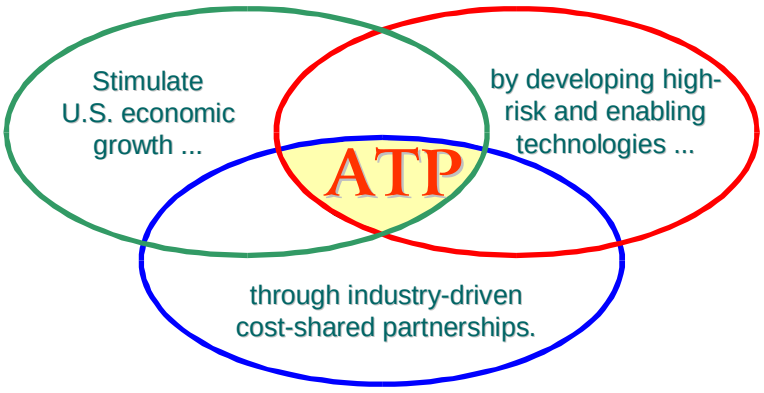
Manufacturing Extension Partnership

Network of centers offering technical assistance and best business practices to the 385,000 smaller manu-facturers in all 50 states and Puerto Rico.

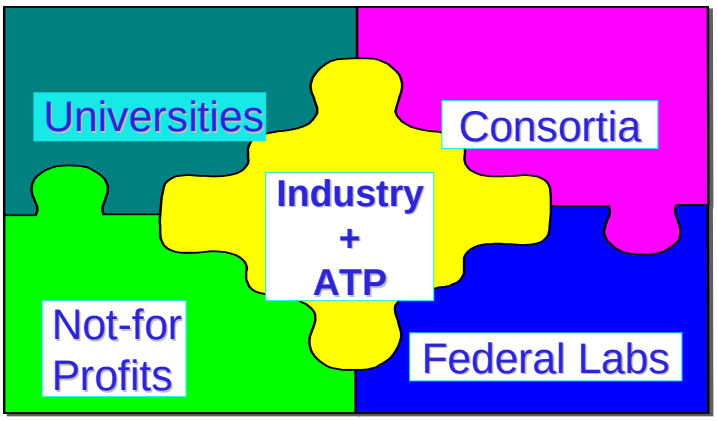
Baldrige National Quality Program

Promotes business performance excellence and quality achievement by U.S. companies.

ATP Mission



ATP Partners



The Alliance Network:
www.atp.nist.gov/alliance/welcome.htm

A Decade of Innovation

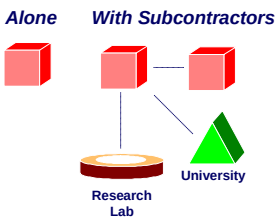
- 4,696 proposals submitted to 42 competitions, requesting $10.1B from ATP
- 581 projects awarded with 1250 participants and an equal number of subcontractors
- 185 joint ventures and 396 single applicants
- $3.6 billion of high-risk research funded
  - ATP Share = $1.8 billion
  - Industry Share = $1.8 billion
- Small businesses are thriving
  - 61% of projects led by small businesses
- Over 150 universities participate
- Nearly 25 national laboratories participate

Two Major Project Selection Criteria

- ***Scientific and Technological Merit (50%)***
  - Innovation in technology
  - High technical risk and feasibility
  - Quality of R&D plan
- ***Potential for Broad-Based Economic Benefits (50%)***
  - Economic benefits
  - Need for ATP funding
  - Pathway to Economic Benefit

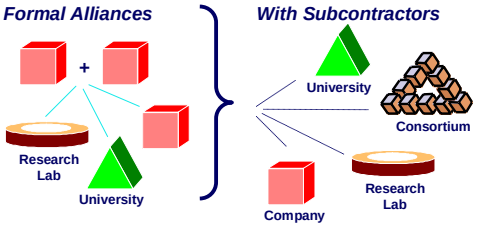
Two Ways to Apply …

As a Single Applicant:



- For-profit company
- 3-year time limit
- $2M award cap
- Company pays indirect costs
- Large companies cost share >60% of total project cost

Or, as a Joint Venture Applicant:



- At least 2 for-profit companies
- 5-year time limit
- No limit on award amount (other than availability of funds)
- Industry share >50% total cost

- ATP encourages teaming arrangements
- Most projects involve alliances

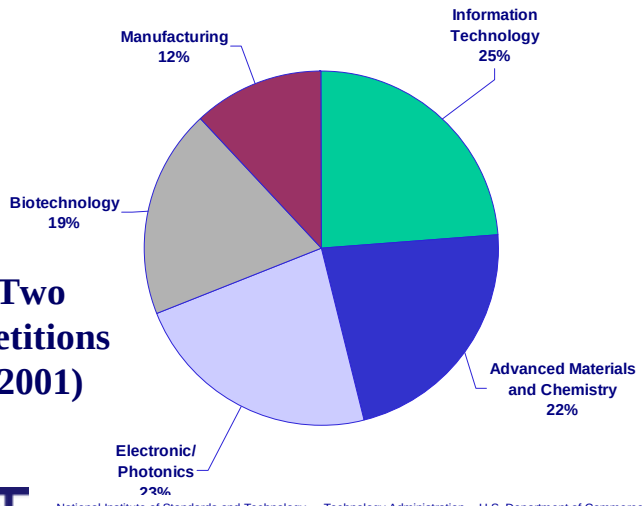
FY2001 Competition Results (to Sept. 2001)

- *261 Proposals Submitted*
- *71 Semifinalists Selected*
- *59 Awards*
  - *13 Joint Ventures*
  - *46 Single Companies*
  - *$164 M ATP Funds; $122 M Industry Funds*
  - *$2.8M average award size*

581 ATP Awards

by Technology Area

(As a Percent of $1,800 M Awarded)



Forty Two Competitions (1990-2001)

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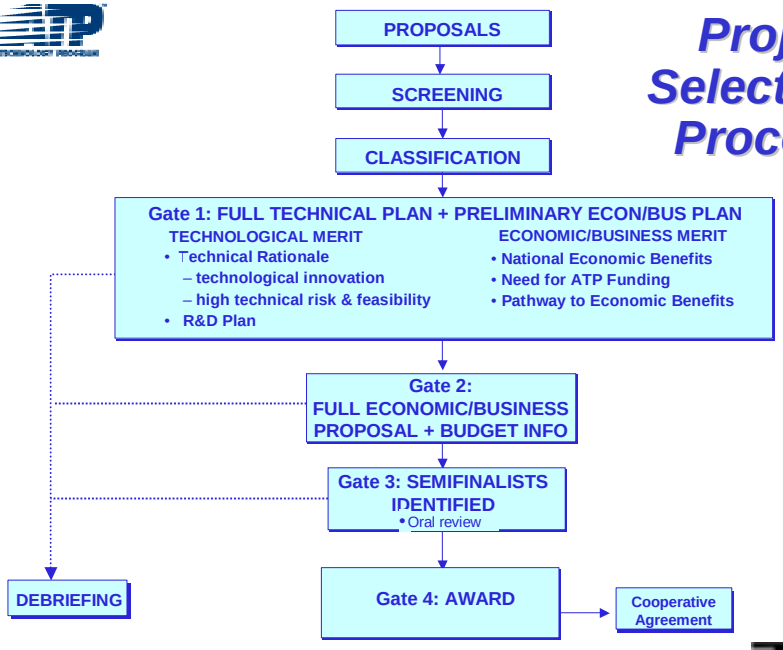
ATP Announces FY 2002 Competition (www.atp.nist.gov)

- **Single Competition open to all technology areas:**
  - A total of $60.7 million is available for 1st year funding of new awards (incl. late awards from 2001)
  - Continual acceptance of proposals until Sept. 30, 2002
  - To be considered for funding in 2002, proposals must be submitted by June 10, 2002
- **Project selection criteria and peer review remain the same**
- **Gated Process- same as for 2001** (see below)

Project Selection Process



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Intellectual Property Provisions

- For-profit companies retain title to intellectual property
- Universities/non-profits may receive payments from royalties; cannot own title
- Companies free to license
- Government reserves right to non-exclusive license for government use only
  - will not disclose proprietary information
  - to date government license never used

Foreign-Owned Company Participation

- U.S.-incorporated subsidiary eligible to participate
- NIST conducts foreign eligibility finding to determine if:
  a) in the economic interest of U.S.; and
  b) parent's country of incorporation affords:
     - U.S. companies opportunities to participate in government-funded programs similar to ATP
     - U.S.companies local investment opportunities
     - Adequate and effective protection for intellectual property rights of U.S. companies
- Refer to "ATP Eligibility Criteria for U.S. Subsidiaries of Foreign-Owned Companies: Legislation, Implementation, and Results"

Chemistry & Process Science Agenda

Cost-effective design methods for environmentally benign routes to materials, chemical building blocks, industrial gases, drugs, food processing etc.

Project Impact Areas:

- **Reaction Design** -- active site design tools and methods to speed development cycle of catalytic and biocatalytic chemistry innovations
- **Process Science** – integration of advanced reaction design and product recovery technologies into cost-effective processes
- **New Materials** -- New catalytic materials, coatings, membranes; control of materials properties at nano-scales
- **High Throughput Methods** -- combinatorial methods for material design, reaction engineering, and processing techniques
- **Efficient Energy Generation** -- New materials and processes for integration into advanced power technologies

ATP Technology Clusters

1993-2001

	Catalysis & Biocatalysis Technologies	Membrane/ Separations Technologies	Fuel Cell Technologies
Active or completed projects:	25*	24**	13
Estimated ATP funding:	$ 98.3 M	$ 47.7 M	$ 37.0 M
Industry cost-share funding:	$ 103.7 M	$ 53.1 M	$ 35.0 M
Total Impact:	$ 202.0 M	$ 100.8 M	$ 72.0 M

*includes 1 cross-cutting issue – Fuel processor for distributed power

**includes 3 cross-cutting issues -- fuel cell membrane

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ATP Technology Cluster
Selective Membrane Platforms

Awardee:	Technology Challenges
Facilichem*	Stable Liquid Membranes: agricultural & biotechnology products
Praxair/WJA	Hydrogen-selective, composite Membranes for H_2 production
Osmonics/Cargill	Solvent & Temp Stable Polymers: seed oils, pharmaceuticals
Engelhard	Controlled Pore Molecular Sieves – one-step purification of natural gas, oxygen enrichment of air streams
Plug Power/ * Polyfuel, Inc., SRI	A highly simplified, CO tolerant PEM Fuel Cell System utilizing advanced components, high temperature membrane *(cross-cutting project)*
MEDAL/Chevron	High-Performance Composite Molecular Sieving Membranes for high separation efficiency of natural gas, industrial gases
Praxair	Develop a novel on-site electrolytic system to separate and compress oxygen from air
New in 2001-	
CombiSep, Inc.	Multiplexed Capillary Electrophoresis for Comprehensive Peptide Mapping
Microcell Corp.	Fabrication of Fuel Cells from Microcell Fibers *(cross-cutting)*
TriboFlow Sep.	Triboelectric Separation of Ceramic Impurities from Powder Metallurgy Alloys

**recently closed*

Osmonics-Cargill-GlaxoSmithKline
NIST-ATP Project

Platform	Target Industry	Chemical Environment
I	Food	Aliphatic solvents
II	Pharmaceutical	Polar solvents
III	Petrochemical	Aromatic, High Temp.

- Platform approach allows for systematic development of materials & applications
- The three platforms cover most solvent environments

Goals-

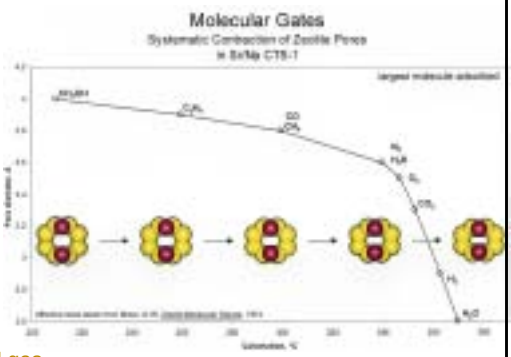
- Develop 2-3 membrane chemistries per platform
- Develop solvent compatible modules, systems
- Develop application intelligence - what membrane to use for what application

5-year, $8.338 million program; Federal share, $3.752 million

Application of Molecular Gate™ Technology To Oxygen Enrichment of Air and Simplified Purification of Natural Gas

Project Objectives:

- Simplified, One Step Purification of Natural Gas
- New processes for oxygen enrichment of air streams
- Develop practical Molecular Gate™ membranes



Outlook:

- High market interest in
  - co-removal of CO_2 and N_2 from natural gas
  - air separation
- Recent ***Nature*** publication, ***412****, 720 (2001),* has generated many suggestions on new applications from around the world

Company: **Engelhard Corp.**
Universities: Cleveland State, U of Massachusetts/Amherst
Start / End: 12/01/99 - 12/31/02
NIST/ATP Funds: $1,800,000
Total Project Cost: $4,500,000

High-Performance Composite Molecular Sieving Membranes

Project Objectives:

- **Develop composite molecular sieving membranes that combine the robustness and processability of polymer membranes with the separation selectivity of molecular sieves. . . reduce processing costs and improve separation efficiency for natural gas, industrial gases and other applications.**

Outstanding Technical Challenge:

- **Materials engineering of the composite membrane**

Impact:

- **Reduce the costs of natural gas processing to the point where sub-quality U.S. gas reserves could be developed.**
- **Reduce production costs of pure nitrogen by 25 to 30 percent for applications such as in-flight inerting of airplane fuel tanks.**

MEDAL/ CHEVRON
Universities: Georgia Tech
Start / End: 11/1/00 - 10/31/03
ATP Funds: $2,695,000
Total Project: $5,401,000

Advanced Electrolytic System for Combined Oxygen Separation and Compression

Project Objective:

- **Develop manufacturing processes and high-pressure components needed for a novel on-site electrolytic system to separate and compress oxygen from air – providing a point-of-use generator capable of delivering high oxygen purity and high delivery pressure at low cost.**

Outstanding Technical Challenges:

- **Choice of materials for the electrodes and the development of a manufacturing process to control their microstructure.**
  - Can porous, thin-film electrodes withstand such high pressures, internal stresses?
  - Can the desired electrochemical properties can be achieved?

Impact:

- **Provide convenient and lower-cost oxygen supplies for industrial and medical uses -- the system could be 70 percent cheaper than traditional distribution methods.**

Praxair, Inc.

Subcontractors: LBNL, Media & Process Techn., Materials Resources Intnl.
Start / End: 11/1/00 - 10/31/03
ATP Funds: $1,982,000
Total Project: $6,006,000

Integration of Membrane Technology and Power Systems

New opportunities for substantial advantages in power density, CO tolerance and water management in fuel cell power systems are driving this important recent trend, as evidenced by:

- ✓ New fuel cell technologies incorporating innovative new membrane systems
- ✓ New membrane-based, hydrogen generation technologies that can be integrated into a fuel cell power system

ATP Technology Cluster
Fuel Cell Power Systems

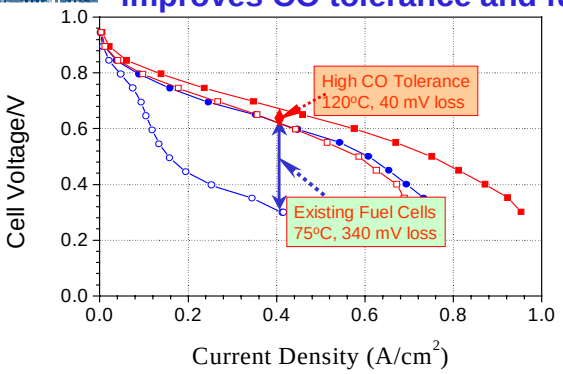
Awardee:	Technology Challenges
Avista Labs*	Modular 2KVA Fuel Cell Power Plant with Live Replaceable, Self-Hydrating PEM Smart Cartridges
Plug Power/ * Polyfuel, Inc., SRI	Standalone fuel cell power systems that can tolerate high levels of CO, operate with standard hydrocarbon fuels
Blasch	Self propagating High Temperature Synthesis of Solid Oxide Fuel Cell Cathode Material
New in 2001-	
MTI Microfuel Cells/ E. I. du Pont	Integrated Hybrid Direct Methanol Fuel Cell/ Electrochemical Capacitor Powerpack
Plug Power	Development of a Highly Reliable and Low Cost Fuel Processing System for Stationary PEM Fuel Cell Applications
T/J Technologies	Hybrid Ultracapacitor/Methanol Fuel Cell Power Paks for Portable Electronics
Microcell	Fabrication of Fuel Cells from Microcell Fibers

*recently closed

Distributed Premium Power Fuel Cell Systems Incorporating Novel Materials and Assembly Techniques

- 3-year, 3-phase, $9.74MM program: 5/99 to 5/02
  - technology development
  - component integration
  - system demonstration
- Key objective:
  To develop a fuel cell system with up to 2,000 ppm carbon monoxide (CO) tolerance
- Key approach:
  High temperature membrane, advanced components

Operating at higher temperature significantly improves CO tolerance and fuel cell performance



Conventional method

- Complex reformer with CO clean-up
- Binary or tertiary catalysts, <200 ppm of CO tolerance
- Current membrane is not designed for high temp

Plug Power's method

- High temp membrane, CO tolerance is significantly improved at >150 ºC
- Advanced components

Significant Accomplishments

- Demonstrated 20,000 ppm CO tolerance
- Achieved more than 5000 hrs stable endurance
- Significantly simplified PEM fuel cell system
  - *Simpler reformer - no CO clean up, no low temp shift*
  - *Simplified thermal management - high temp operation*
  - *Simplified water management - water balanced system*
  - *Simpler system integration - fewer parts, transient capable*

Integrated Hybrid DMFC/ EC Capacitor Powerpack

Project Objective:

- **Develop a novel, long-lived, refuelable, batteryless power source for portable electronic devices by combining a miniaturized direct methanol fuel cell with an electrochemical capacitor and a microfluidic system.**

Approach:

- **The technology combines an advanced direct methanol fuel cell with an electrochemical capacitor to meet peak power needs, and a microfluidic system to handle the recirculation of water. The proposed 1-Watt hybrid system would provide the same power as a typical rechargeable lithium battery in the same space, but with a greatly improved life. The time between recharging/refueling) of a cell phone would extend to a full month.**

Impact:

- **This device would provide a new power source for mobile electronics, which allows much longer run times and with a higher energy density than for lithium batteries. For mobile phones alone, the market could reach $9 billion by 2010.**

MTI/ du Pont
Start / End: 10/1/01 - 9/30/04
ATP Funds: $4,662,456
Total Project: $9,343,002

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Fabrication of Fuel Cells From Microcell Fibers

Project Objective:

- **Develop a new fuel-cell technology based on a novel microfiber membrane structure, with revolutionary advances in increased power density.**

Approach:

- **In a microcell, gaseous or liquid fuel passes through a channel within the fiber. A critical inner current collector is encapsulated by a porous membrane separator, which contains an electrolyte within its pores. The inner and outer shell of the fiber is coated with an electrocatalyst. Fuel cell prototypes of 1 to 5 kilowatts, with a power density of at least 400 watts/liter, will be fabricated from modular assemblies of microcell fibers.**

Ultimate cost target:

- **Below $100 per kW**

Impact:

- **These new microfiber fuel cells could open up large markets in stand-alone power for homes and businesses and could eventually offer economic vehicuar power.**

Microcell Corporation
Start / End: 11/1/01 - 10/31/04
ATP Funds: $1,998,000
Total Project: $2,402,000

High-Temperature Hydrogen Selective Membrane Platforms

Project Objectives:

- **Develop an economical hydrogen-generation system and high temperature hydrogen separation membrane platform. The generation system combines a novel palladium-based, high temperature, hydrogen-selective membrane with a catalytic reactor.**

Outstanding Technical Challenge:

- **To produce a hydrogen separator with an extremely thin defect-free membrane capable of stable operation at high temperature**

Recent Success:

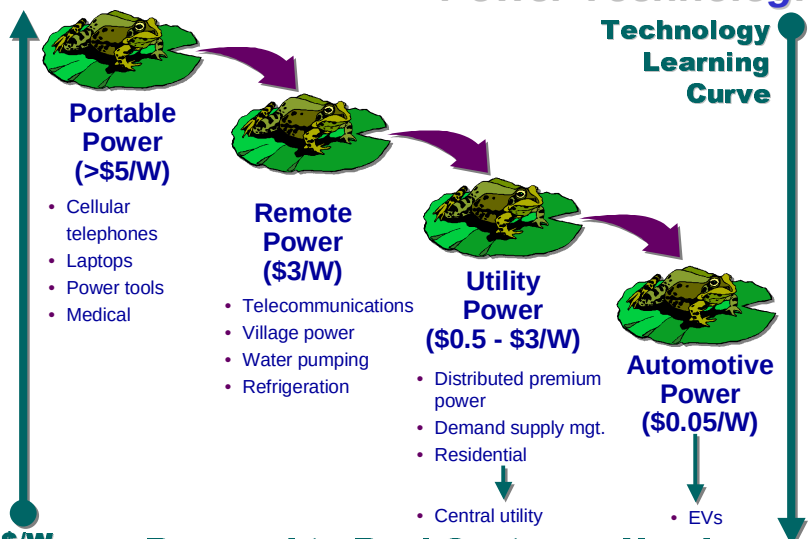
- **Commercial hydrogen purifiers for small-scale production of ultrapure hydrogen at high fluxes.**

Outlook:

- **To develop a pilot scale membrane reactor for the production of hydrogen that can be used to power fuel cells**

Praxair/ WJA
Universities: Tufts U
Start / End: 2/1/99 - 1/31/04
ATP Funds: $1.515 M
Total Project: $3.082 M

Leap Froging to Tomorrow's Power Technologies



Respond to Real Customer Needs

Intramural Programs at NIST-

Infrastructure for Development of Selective Membrane Platforms

Characterization tools for new membrane materials and structural forms that provide exquisite control of chemical selectivity, while maintaining high flux (productivity)

- *Multicomponent transport – robust and accurate ATR-FTIR flow system*
- *Pore metrology – AFM techniques and hardware*
- *Membrane database – Internet accessible database*

New membranes for high-throughput purification of circular DNA

- *The density and physical structure of the sites for trapping circular DNA in membranes can now be controlled..*
- *Design and fabrication of a membrane module with integrated electrodes; performance tests using plasmid DNA samples.*

Chromatographic and Electrophoretic Techniques to Investigate Chiral Recognition

- *Slight structural differences among families of analytes result in disparities in chiral recognition and provide insight into the structural features needed for successful chiral resolution with cyclodextrin-based chiral selectors*

Infrastructure for Development of Selective Membrane Platforms

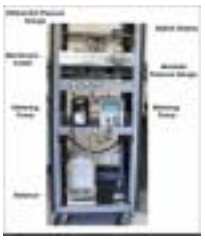
Multicomponent Membrane Transport Metrology

FTIR-ATR techniques and data reduction for pre-made films and membranes

H_2O and acetone diffusion in PP used as a model system for hydrophobic materials—accounted for a surface mass transfer resistance for H_2O

Membrane Pore Metrology

initial design and construction of liquid-liquid porosimetry apparatus based on flow control versus diff. pressure control



Membrane Database

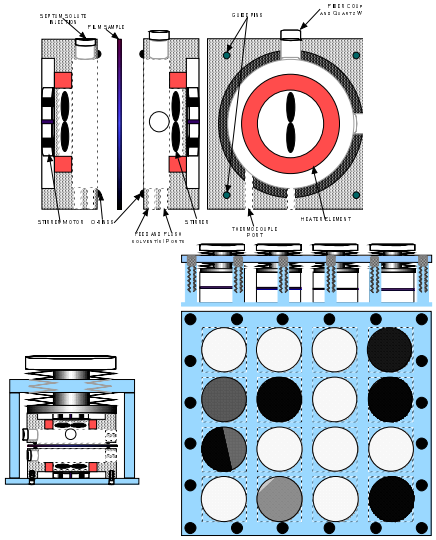
created relational database for pressure-driven liquid separations

NAMS-sponsored R&D project
Standards for UF Membranes

High-Throughput Screening of Molecular Transport through Films, Membranes, and Nanostructures

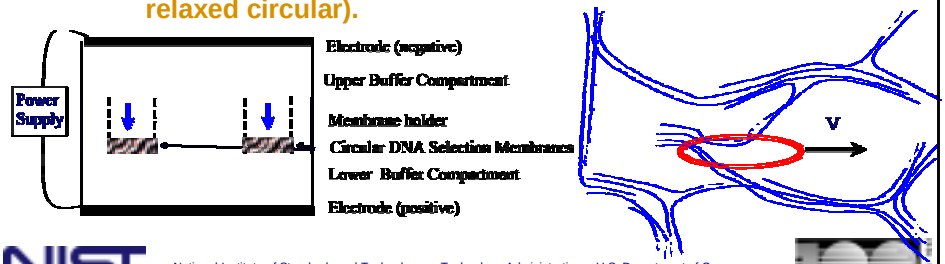
Design, fabricate, construct and demonstrate a multi-well apparatus including:

- transport cells,
- fluid manipulation hardware,
- fiber-optic based fluorescence excitation and emission detection hardware,
- data acquisition and control hardware and software,
- data analysis and modeling, and
- families of probe molecules (e.g., PEG-modified dendrimers

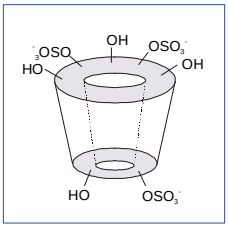


Preparative Separations of Circular DNA Using Membranes

- **Electric fields are used as driving force for rapid separations.**
- **Gel-filled membranes can be formulated for specific separations.**
- **Density and physical structure of "circle traps" can be controlled.**
- **Separations tuned by electric field strength and membrane.**
- **Specific for circle size and topology (supercoiled and relaxed circular).**



Use of Chromatographic and Electrophoretic Techniques to Investigate Chiral Recognition



Simplified structure of cyclodextrin

Emphasis on use of various modified cyclodextrins as chiral selectors

Focus on enantioselective separations of pharmaceutical compounds

Families of structurally related compounds used as probes of enantioselectivity

Goals:

- **Improve our understanding of the mechanisms responsible for chiral recognition**
- **Utilize this knowledge to facilitate development of chiral separation methodology**

Role of NIST/ATP's Intramural Program

The Intramural Program within NIST provides critical infrastructural support of ATP's extramural (external) portfolio, at the same time broadening and enhancing NIST's competency in its overall support of U.S. industry.

ATP's Extramural Program - Broad Economic Benefits Sought

- **Enable U.S. manufacturers to be Value Leaders with new and higher performance membrane/ separations systems**
- **Enable U.S. manufacturers to be Cost Leaders, providing greater than 20% cost reduction in a core-process separations operation**
  - Improved energy economics, pollution abatement
  - Reduced byproduct and waste
- **New business opportunities, spin-offs**
  - ready extension of new membrane/separations platforms into new applications
  - New, improved routes for delivering specialty chemicals that will enable creation of new or higher performance products

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1999 ATP Workshop on Membrane/ Separations Technology

Comment on Theme-

Critical Barriers • Multi/cross-disciplinary

Unique, multidisciplinary teams are required to address critical barriers/ technology hurdles to wider application of membrane technology. ATP can help facilitate the creation of these teams and support the most critical phase of their efforts to meet these high-risk challenges and bring new, enabling technology for entire industry sectors to the marketplace.

For Info on ATP and to Join Our Mailing List . . .

- Call toll-free: 1-800-ATP-Fund (800-287-3863)
- Fax your name and address to: (301) 926-9524
- Send an e-mail message to: ***atp@nist.gov***
- Visit ATP's website: ***www.atp.nist.gov***

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